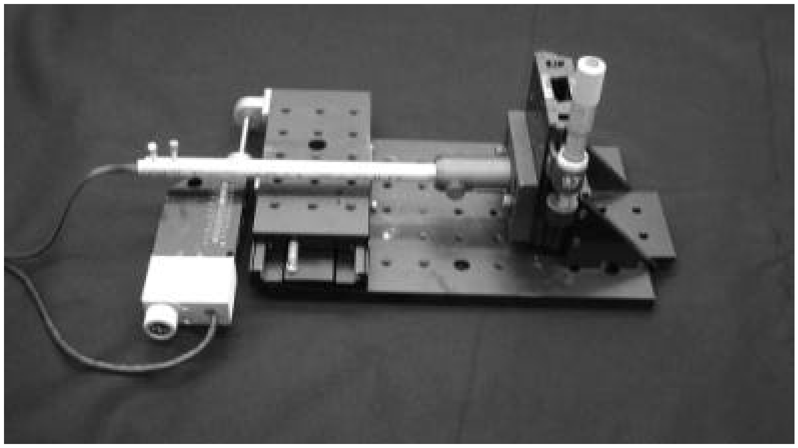

Ultra-Violet/Ozone Gradient Instrumentation

Specifications and Operation Guidelines

For gradients in
UV/Ozone Exposure · Surface Energy · Chemical Functionality
Crosslinking

Combinatorial Methods Center

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Last Update: September, 2002 (Fasolka)

1. Purpose and structure of this document

This document is provided by the NIST Combinatorial Methods Center as a guide for constructing and operating an instrument for creating gradients in Ultra-Violet (UV)/Ozone exposure on planar specimen surfaces. First, the basic principles of this instrument will be described. Next, the components of the instrument and schemes for its construction are supplied. Here, the discussion is based upon the specific components and design of the NCMC device (see disclaimer, page 1). Next, guidelines for operating the instrument and some basic applications are outlined, including notes on computer control/automation and calibration.

2. Principle of the NCMC UV/Ozone Exposure Gradient Instrument (UVOGI)

The NCMC UVOGI is based upon simple concepts. As seen in Figure 1, a UV light source illuminates a planar specimen through an aperture. The specimen is translated under this aperture via a motorized stage. Acceleration (a) of the translation stage provides the means for producing an exposure gradient. Tailoring the acceleration function f(t) (e.g. through computer control specifies the depth and steepness of the gradient.

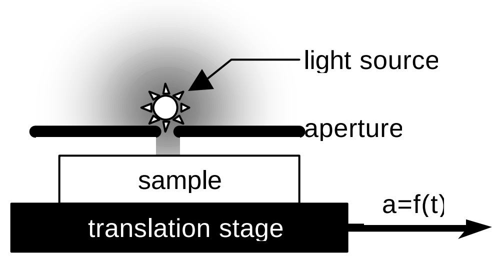


Figure 1: UVOGI Principles

3. Design of the NCMC UVOGI

Figure 2 illustrates the UVOGI in plan view. Figure 3 shows a side view. Device components are labeled (A-F) and described below.

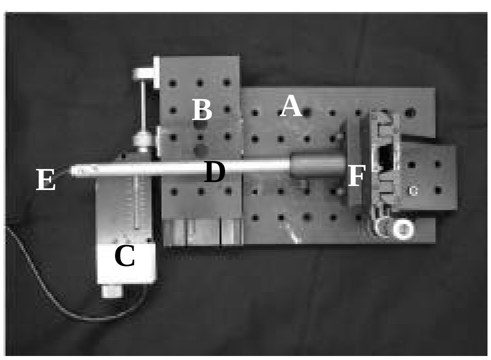


Figure 2: Plan View

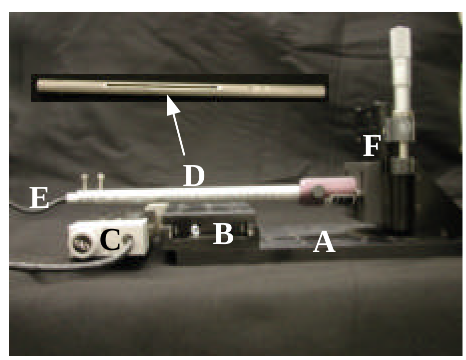


Figure 3: Side View

- **A) Base.** Optical platform with ¼-20 bolt taps. (Newport)
- **B) Linear Translation Stage/Sample Platform.** (Newport 443 Series)
- **C) Stage Actuator.** Step-motor actuator with 4.5 cm total travel. Computer controlled via manufacturer supplied driver card. (Newport 850F series)
- **D) Slit Aperture/Lamp Housing.** 10” long aluminum piping with ½” OD, 3/8” ID. Aperture (see inset, white arrow) is a machined 2 mm wide slit. The

aperture/housing is mounted to the z-translation stage (F) via an optical post mount (Newport).

E) UV light source. See also Figure 4A. The NCMC uses two UV sources. 1) Low Pressure Hg vapor wand lamp with fused silica envelope. The primary emission is 254 nm. A secondary emission at 185 nm dissociates molecular oxygen in the presence of ozone. (120V, 30mA; Jelight, part # 78-2046-7). Approximately 8” long, the lamp is inserted into the housing (D) and secured with two setscrews. The lamp is powered via a manufacturer supplied power source (Jelight, part # PS-2000-20). 2) Low pressure Hg vapor wand lamp with glass envelope and phosphor coating. Emission is 285 nm and no ozone is created (Jelight, part #84-285-7).

F) Z-translation stage. Controls sample/aperture distance via a micrometer. Mounted onto base via angle bracket (Newport 443 Series).

Figure 4 details the light source and some the aperture appliances in use in the NCMC.

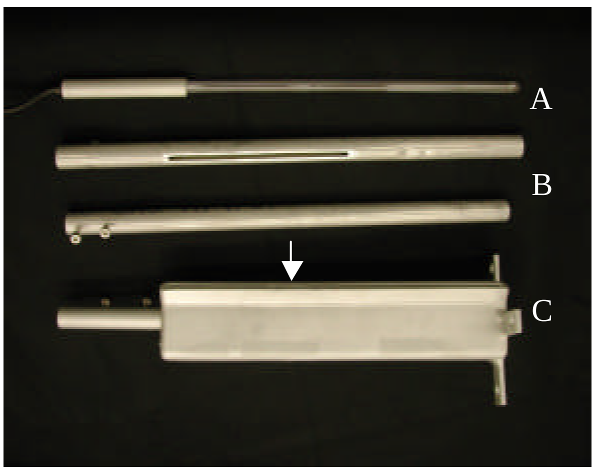


Figure 4: UVOGI source and aperture appliances.

A) UV wand Lamp. A fused silica envelope is *required* if ozone generation is desired.

B) Aperture/Lamp housing. Top image shows slit aperture. Bottom image shows setscrews for securing lamp.

C) “UV only” aperture appliance. Designed to keep the lamp > 5 mm above the sample so that it is exposed to UV but not ozone. The slit aperture is at the end of a thin flange and is 1mm wide (see white arrow). The lamp is secured with setscrews on the left. The appliance is attached to the z-translation stage via built-in brackets (see right side of Figure 4C).

4. UVOGI Use Guidelines

The NCMC UVOGI is operated through a Windows graphical interface built with Labview Software (verson 6i, National Instruments). More information about the specific subroutines used and how they work can be found in Appendix 1. Labview requires a National Instruments PCI card to actually drive instrumentation (here, IMAQ PCI-1411).

Figure 5 shows a snapshot of the UVOGI graphical interface with some typical operational parameters. The buttons and dialogue box on the left allow the stage to be reset and moved to an appropriate starting position. Operational parameters are inserted into dialogue boxes at the top left. There are four input parameters. The value “δmax” (L) specifies the total distance the stage will travel in mm. The “step size” (d)

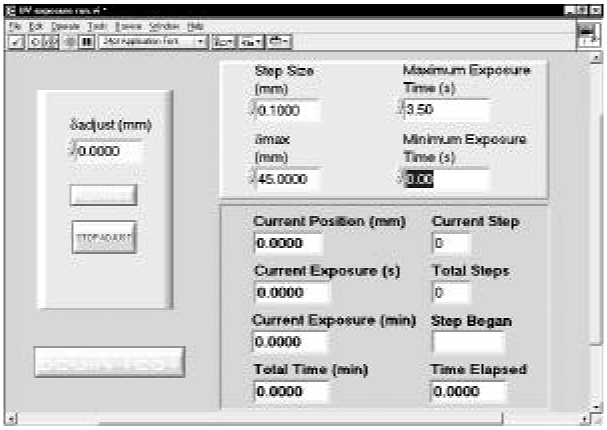


Figure 5: Snapshot of software interface

gives the increment of stage movement in mm. Accordingly, there are $N=L/d$ steps during the total exposure. After each stage movement step, the stage waits for a specified exposure period. Gradients in exposure are created by incrementally increasing this period at each step. The exposure time function is linear. It is defined through the two parameters, “maximum exposure time” (E_{max}) and “minimum exposure time” (E_{min}), each in seconds. Thus, at the n^th^ step, the exposure period is $n((E_{max}-E_{min})/N)$.

These parameters allow for the gradient depth and slope to be tailored. The exposure function also depends upon the aperture width (*w*, in our case 2mm). When $d<<w$, a smooth gradient is created, with a nominal exposure profile like that shown in Figure 6. When *d*» *w*, a step-like exposure function is created. When $d>>w$, widely spaced regions (*w* wide) of increasing exposure are formed. Note, however, that the time *between* steps is not included in these calculations. This can be easily corrected by considering the velocity of the actual actuator used.

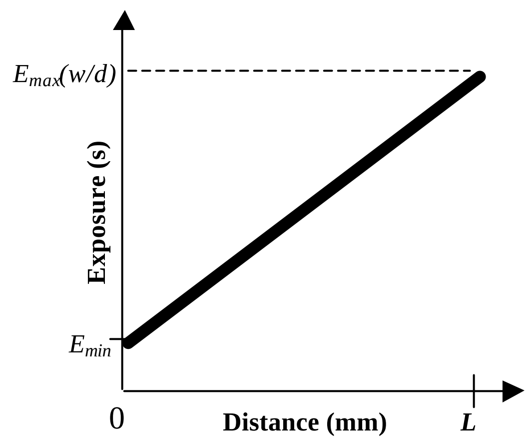


Figure 6: Exposure function for a smooth gradient

A typical UV/Ozone exposure gradient is generated as follows:

1) The UV source is powered. Typical UV lamps take 5-10 minutes to reach their maximum intensity. To avoid sample exposure during subsequent set-up steps, the aperture appliance is raised to its maximum position via the z-translation micrometer. Alternatively, the aperture can be temporarily rotated away from the stage or covered in some manner during the power-up period.
2) The sample is installed on the stage.
3) Using the software control, the stage is moved to the starting position.
4) The exposure function is defined using the dialogue boxes.
5) Using the z-translation micrometer, the aperture is lowered to a distance *h* (typically ≈0.1mm) above the sample surface.
6) The gradient exposure is initiated using the software control. In the NCMC instrument, the software displays an estimate of the time required for the procedure to finish. This is important, because the NCMC design does not include means to automatically stop UV exposure after the procedure is complete. Accordingly, to

avoid over-exposure of the end of the gradient, the source must be turned off manually.

5. Application Examples

Variable UV/Ozone exposure using UVOGI can result in gradients in a number of specimen properties. Often, the aperture/sample distance (*h*) during exposure determines what kinds of gradients are produced. Ozone generated by the UV source depletes quickly. To ensure ozone exposure, the source must be positioned within 5 mm of the sample surface. If only UV exposure is desired, *h* must be kept > 5mm or an ozone free source must be used. The different aperture appliances designed for use with the NCMC UVOGI enable a variety of sample/surface distances while maintaining adequate source collimation. Next, a few basic application examples will be discussed.

Surface Energy/Chemical Functionality Gradients
The NCMC strategy for creating surface energy gradients (SEGs) requires ozone exposure. Accordingly, the slit source appliance (Figure 4B) is used, since it allows *h* < 5 mm. The basis of SEG generation is an ozone-sensitive self-assembled monolayer (SAM) applied to the sample surface. The SAM (e.g. n-octyldimethylsilane, Gelest Inc.) gradually converts to oxygen-containing species as ozone is applied. For alkyl chain SAMs this conversion increases the local surface energy (more hydrophilic). Example operating parameters for generating an SEG are: h=0.1 mm, d=0.1 mm, E_{min}= 0 s, and E_{max}=4 s. On n-octyldimethylsilane treated surfaces, these parameters create a gradient exhibiting water contact angles (a measure of surface energy) from 95° to < 25°. Figure 7 shows an example gradient. Of course, results will vary depending upon the SAM used. Ozone can also be used to create chemical functionality gradients (CFG). In the above example, the SEG is also a CFG as oxygen-containing moieties are being added to the surface. Using the UVOGI to create a CFG requires an UV or ozone sensitive chemistry with which to work. Suppliers of SAM/surface modification chemicals offer a wide range of products that could be modified by the UVOGI or like devices.

Figure 7: Surface energy gradient on silicon wafer produced with UVOGI and the NCMC technique. The decreasing water contact angle is evidence of the gradient.

UV-Exposure, Crosslinking and Photo-polymerization gradients
In some applications UV radiation exposure is desired, but without the degradation effects of ozone. In such cases, *h* must be greater than 5 mm. This can be accomplished using the slit aperture appliance, but substantial widening of the exposure footprint will result. If a collimated source is necessary, construction of an appliance similar to that in Figure 4C is recommended. Alternately, use an ozone-free UV source as described above. In general, UV-exposure (e.g. “aging”) gradients involve much longer illumination times. For example, in NCMC studies of UV degradation of polymer/metal interfaces, E_{max} is on the order of 300 s and d=0.1 mm, resulting in an

exposure routine that takes a few hours to complete. Photo initiated crosslinking (see Figure 8) and polymerization gradients require a somewhat shorter time scale. Ultimately, however, this depends upon the exact chemistry and formulation used.

Figure 8: Step-like gradient in UV crosslinking of polyvinylcinnamate.

6. Other Notes

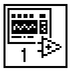
- A variety of other aperture appliances are possible. For example, single holes or rows of holes can be machined into the lamp housing, so exposed stripes can be formed.
- Other light sources, with a variety of emission spectra can be purchased from several manufacturers.
- Constant surface energy surfaces can be made through a flat exposure function ($E_{max}=E_{min}$).
- For some applications, it is desirable that the exact UV dosage is known. This calibration involves a precise measurement of the lamp output. While the NCMC does not provide such calibration services, NIST is the world leader in light intensity measurements. Interested parties should contact the Optical Technology Division at NIST.
- In NCMC studies, surface energy gradients are measured/calibrated via contact angle measurements using an automated contact angle measurement system (Kruss, model G2-Mk4). The surface energy is calculated from contact angle measurements from two different fluids, typically water and a non-polar solvent.

Appendix: Computer Automation Software

The NCMC UVOGI is driven by a graphical interface built using Labview software (version 6i, National Instruments) using both custom written routines and library functions. Labview hardware control is mediated through a PCI cards purchased from National Instruments (e.g. IMAQ PCI-1411). Below, basic information on the software structure and the routines used is provided. UVOGI automation routines are available to NCMC members and collaborators through the NCMC website (www.nist.gov/combi) or by contacting the NCMC at NCMChelp@nist.gov. A Labview software license is required for their use. Furthermore, the NCMC does not provide any warrantees, guarantees or extensive technical support for software it offers.

UV exposure run.vi

Connector Pane



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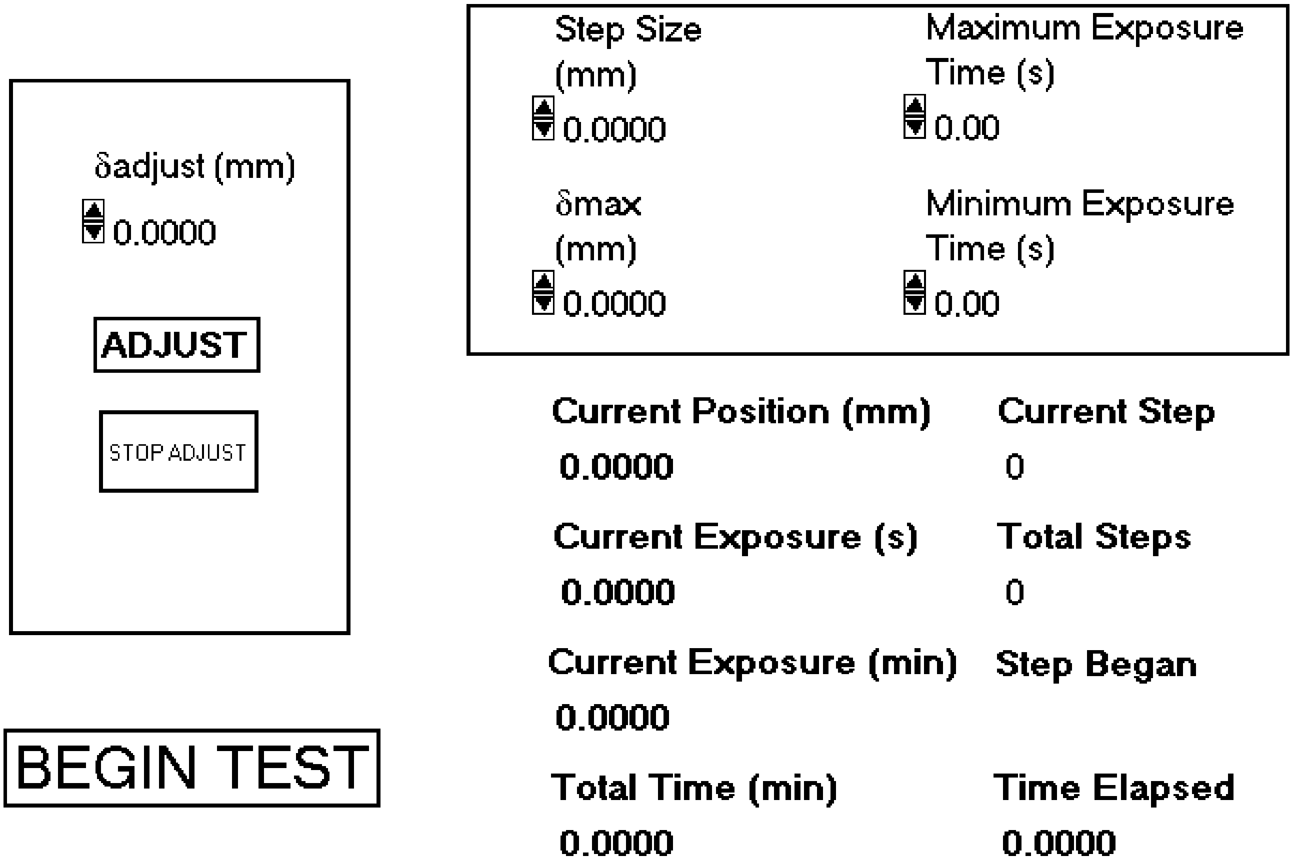
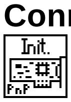


Initialize system.vi

Connector Pane



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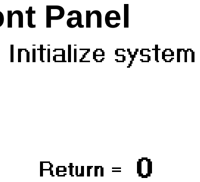

Error, read & display if any.vi

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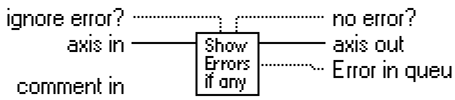

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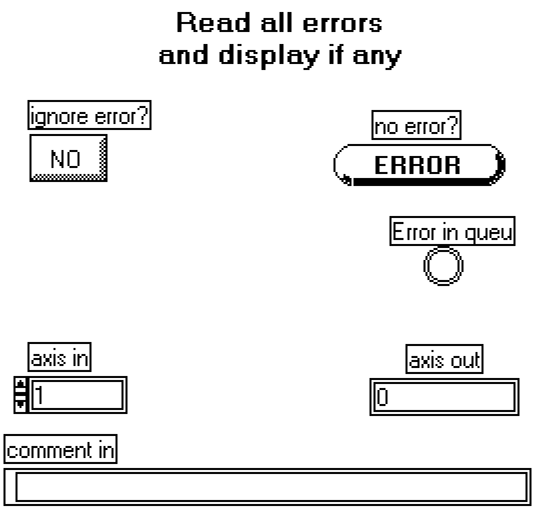
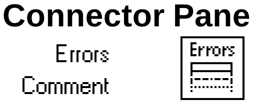


Error, display list.vi

Connector Pane



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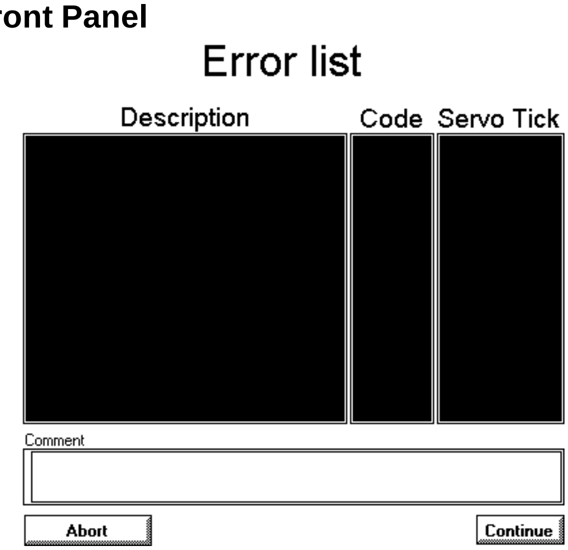

Error, read all.vi

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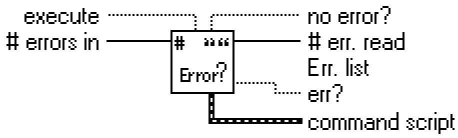

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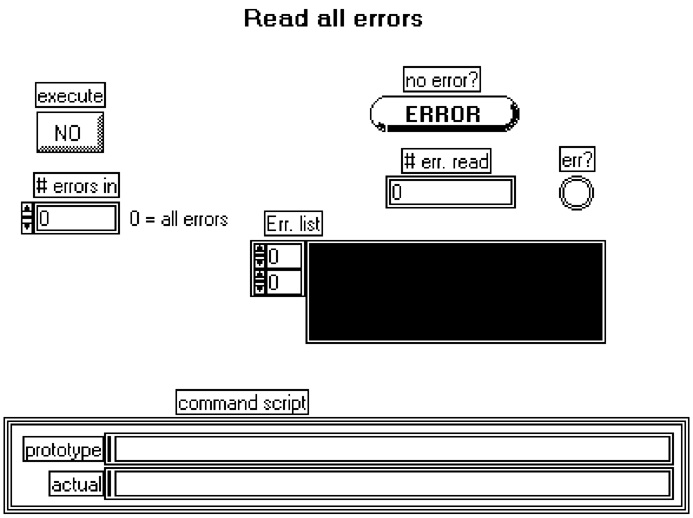
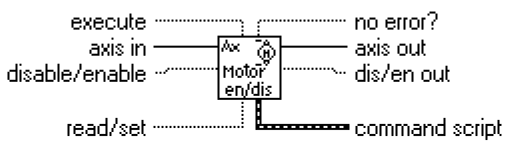


Motor, enable/disable.vi

Connector Pane



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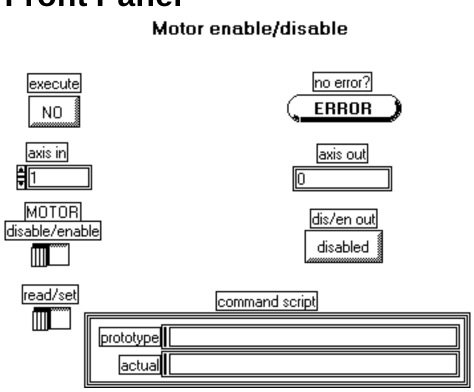
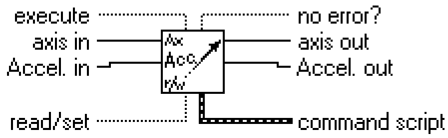


Acceleration.vi

Connector Pane



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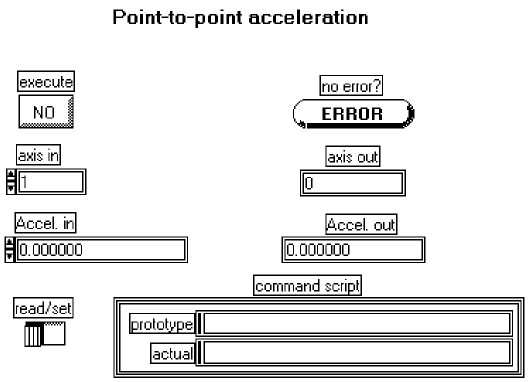
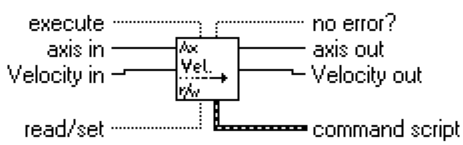


Velocity.vi

Connector Panel



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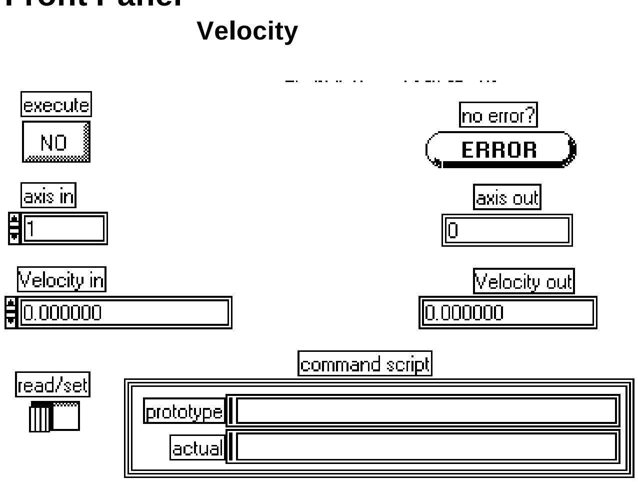
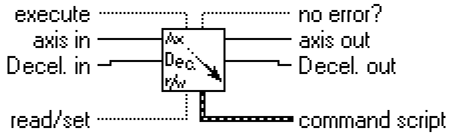


Deceleration.vi

Connector Panel



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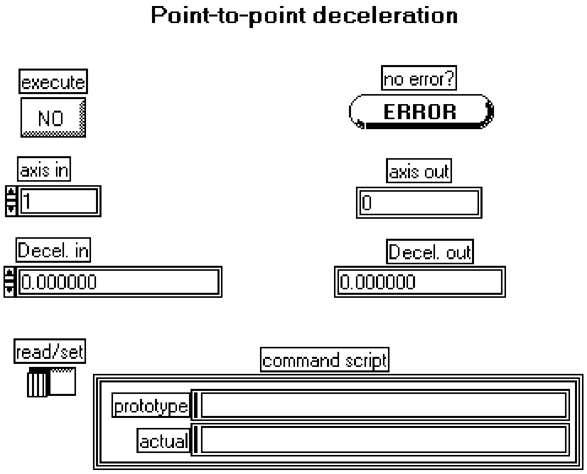
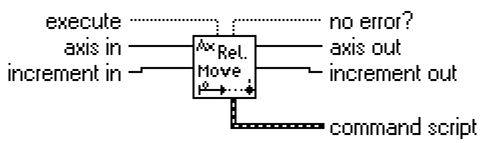


Move to relative position.vi

Connector Pane



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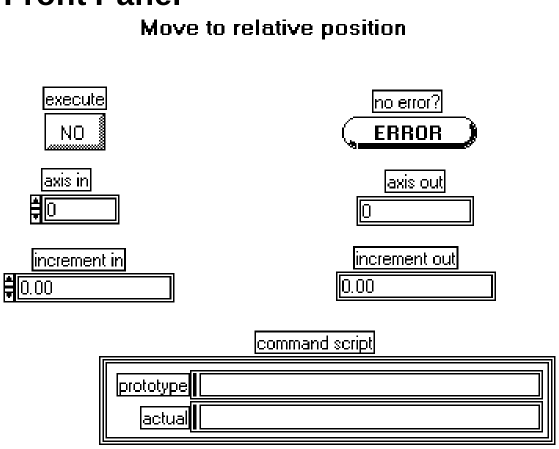
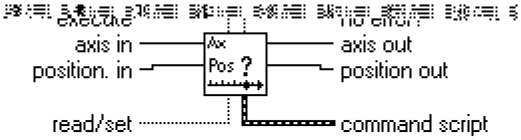


Position.vi

Connector Pane



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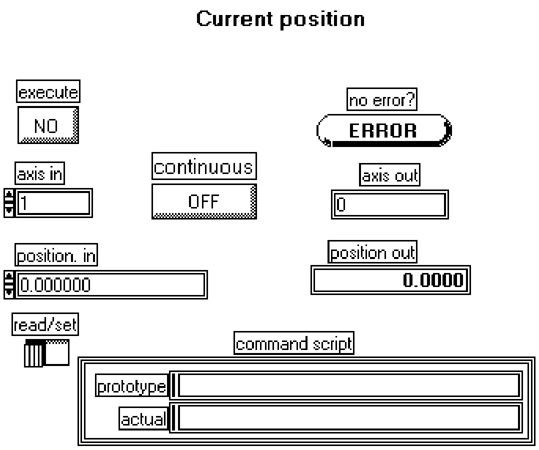
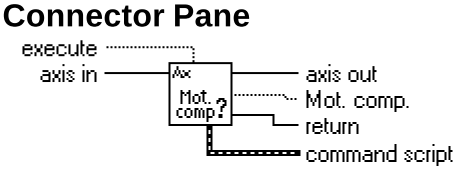


Motion complete.vi

Connector Pane



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